

a silicon alkoxide are initially reacted and this reaction product is then, in the presence of sufficient water, converted to a soluble, substantially linear, further polymerizable, phosphoro-siloxane or, perhaps, more accurately, phosphoro-organo-siloxane, which siloxane is then further polymerized by cross-linking in the presence of sufficient water to create a homogeneous gel material which is subsequently converted to the desired oxide product.

EXAMPLE 1

Into a beaker there were added 1,440 grams (6.92 moles) of ethyl orthosilicate and to this there was then added 700 ml of 96 percent ethanol, thereby producing a single phase solution. To this solution there was added with agitation about 147 grams of an 85.3 weight percent aqueous solution of ortho phosphoric acid. A reaction ensued and there remained a clear solution of the soluble reaction product. To this solution there was then slowly added with stirring about 472 ml of distilled water with heat being liberated and the temperature rising to approximately 65° to 70° C. At this point there was a clear, somewhat viscous solution of the soluble, further polymerizable, substantially linear, phosphor organosiloxane. This solution, after allowing it to stand for a period of approximately 15 minutes, cross-linked and formed a homogeneous gel. The gel was then dried for about sixteen hours at 60° C. in an oxygen ventilated oven, then the temperature was raised to about 110° C. and held there for four hours. After this, the material was heated and held at a temperature of about 200° C. for about four hours and then was allowed to set overnight, that is, a period of about sixteen hours, at a temperature of 300° C. after which time there was produced a jet black product which weighed 553 grams.

This black product is then further heated to a temperature of about 1830° F. (about 1000° C.) for about 18 hours, whereby it converts to a white, particulate mass having a P₂O₅ content of about 16.4 weight percent. This white mass is then passed through a 100 mesh screen (U. S. Sieve) and about 333 grams of the phosphoro siloxane powder as prepared above, are then mixed with about 10 grams of a conventional binder (polyethylene glycol, manufactured and sold under the trade name CARBOWAX 20 M) and approximately 100 cc of acetone. A portion of the acetone is allowed to evaporate until a slurry on the order of the consistency of tooth paste is formed and this slurry is then granulated by passage through an 8-mesh screen. The granulated material is allowed to sit overnight to further complete acetone evaporation and this material then pressed into a disk of about 5.1 inches in diameter with a hydraulic pressure of about 50 tons. This pressed green disk is then sintered by conventional heating techniques. In this instance, the disk first heated to about 500° F. and held there for about one-half hour and then gradually heated to about 1830° F. and held there for about one hour and then it is finally heated to approximately 2280° F. over a period of time of about two hours and held at that temperature for about 15 minutes. The disk is then gradually allowed to cool at room temperature.

This disk is then used as a target material to sputter coatings onto silicon chips and platinum substrates using conventional RF sputtering techniques. In this specific instance a low energy unit such as that supplied

by Consolidated Vacuum Corporation as their type AST-100 unit employing their AST-200, RF amplifier, is used. Excellent high quality films will be produced on silicon chips and platinum substrates using equipment of the type indicated above at an Argon pressure of about 5 microns with 700 watts of power at a frequency of 13.56 mHz.

EXAMPLE 2

The procedure of Example 1 is followed except there is employed anhydrous ethanol and approximately 500 ml of distilled water is added subsequent to the addition of the phosphoric acid. The homogeneous gel which is produced is then thermally degraded and material made in this manner is converted to a substantially unitary body as described above. This body when sputtered under the conditions indicated above produces substantially identical results.

EXAMPLE 3

Into a beaker there is then added about 172 grams of methyl triethoxysilane. To this there is then slowly added with agitation about 24.3 grams of an 85.3 weight percent ortho phosphoric acid solution. A reaction ensued as evidenced by the liberation of heat and after several minutes, a viscous, clear solution of the phosphoro-silicon reaction product was produced. To this clear, viscous solution with stirring, there was slowly added approximately 75 ml of distilled water. After the addition of the water there was a clear solution of a soluble, substantially linear, further polymerizable phosphoro-organosiloxane material. After allowing the solution to stand for approximately three to five minutes, the material cross-linked and produced a homogeneous uniform gel. Gels produced in this manner, when treated in the manner recited in Example 1, produce substantially identical results when they are subjected to sputtering applications as therein set forth.

While the above describes the present invention and enables one skilled in the art to make and use same, it will, of course, be apparent that modifications are possible which, pursuant to the patent laws and statutes, are comprehended within the scope of the present invention.

I claim:

1. The method of producing a homogeneous oxide product consisting of phosphorus and silicon oxides which consists of:

- a. directly reacting phosphorus pentoxide, phosphoric acid, phosphorous acid or mixtures thereof with
 - (1) a compound of the formula $\text{SiX}_n\text{Y}_{(4-n)}$, wherein n is 1, X is phenyl or an alkyl having one to six carbon atoms, and Y is OR wherein R is a C₁—C₆ alkyl or
 - (2) a compound as set forth in (1) wherein n is 1 in the presence of an effective solution forming amount of an organic solvent, or
 - (3) a compound of the formula set forth in (1) wherein n is 0 in the presence of an effective solution forming amount of an organic solvent, or
 - (4) a compound of the formula

